NASA TECHNICAL
MEMORANDUM

NASA TM X-53208

FEBRUARY 15, 1965

NASA TM X-53208

EFFECT OF LIQUID NITROGEN DILUTION ON LOX IMPACT SENSITIVITY

by C. F. KEY AND J. B. GAYLE
Propulsion and Vehicle Engineering Laboratory

NASA

George C. Marshall Space Flight Center, Huntsville, Alabama

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C. F. Key and J. B. Gayle
George C. Marshall Space Flight Center
Huntsville, Alabama

ABSTRACT

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An experimental investigation was carried out to study the decrease in reactivity of materials with liquid oxygen (LOX) that is caused by dilution of the LOX with liquid nitrogen (LN_2). A wide range of materials was selected for testing, each of which previously had been shown to be sensitive to impact in LOX. Tests were made with the ABMA LOX Impact Tester using LOX/LN_2 mixtures ranging in concentration from 20 percent LOX in LN_2 to pure LOX. The results showed that relatively large proportions of LN_2 were required to effect an appreciable decrease in reactivity; however, all materials tested were insensitive to impact at 10 kg-m in liquid air.

AUTHOR

NASA-GEORGE C. MARSHALL SPACE FLIGHT CENTER

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SUMMARY

An experimental investigation was carried out to study the decrease in reactivity of materials with liquid oxygen (LOX) that is caused by dilution of the LOX with liquid nitrogen (LN_2). A wide range of materials was selected for testing, each of which previously had been shown to be sensitive to impact in LOX. Tests were made with the ABMA LOX Impact Tester using LOX/LN_2 mixtures ranging in concentration from 20 percent LOX in LN_2 to pure LOX. The results showed that relatively large proportions of LN_2 were required to effect an appreciable decrease in reactivity; however, all materials tested were insensitive to impact at 10 kg-m in liquid air.

INTRODUCTION

Many materials in contact with LOX constitute fire and/or explosion hazards when subjected to impact, shock, heat, or other forms of energy; organic materials are especially hazardous under these conditions. Although the degree of hazard is decreased when LOX/LN_2 mixtures are substituted for LOX, evidence of sensitivity has been noted for some materials which were tested with mixtures containing 30 percent LOX by weight. However, conclusive evidence to prove that no hazard exists even with mixtures containing only 20 percent LOX (liquid air) has not been obtained.

A previous investigation (ref. 1) indicated that there is a small but finite probability of occurrence of a catastrophic reaction if damaged LH_2 insulation is subjected to a suitable stimulus during or subsequent to LH_2 hold. This occurs because air is condensed within the damaged insulation and, subsequently, may be enriched in oxygen by reevaporation and condensation processes.

The possibility of condensation of liquid air on engineering materials is not limited to LH_2 insulation. Moreover, the probable extent of enrichment of condensed air by reevaporation and condensation processes is difficult to assess, either experimentally or analytically. Therefore, an experimental investigation of the effects of LN_2 dilution on the LOX impact sensitivity of selected engineering materials was made to obtain additional information on this problem.

Most of the samples used for this study were prepared by the Non-Metallic Materials Branch of this division.

EXPERIMENTAL

Test Method

The apparatus used for all of the tests reported herein was the ABMA LOX Impact Tester. The mechanical features and operational details of this tester have been described comprehensively in other reports (ref. 2) and will not be repeated herein. In principle, this test involves dropping a standard plummet of known weight (9.04 kg) from known heights (up to 1.1 meters) under near-frictionless conditions. This plummet strikes a pin which is resting on a layer of the material being tested in the bottom of an expendable aluminum alloy cup. The remainder of the sample cup is filled with the test mixture. During a test, a material capable of reacting with the test mixtures will explode and/or flash brilliantly. The highest energy level that is withstood by a given material without any indication of sensitivity in 20 trials denotes the hazard associated with the material under test when it is used in LOX systems.

Sample Preparation

All metals and elastomers were tested in the form of 11/16-inch diameter discs. Composite insulations and foams were tested as 1/2-inch squares. Type 347 stainless steel inserts were used as false bottoms for the sample cups. This technique was necessitated by the early discovery that some hard materials could give a false indication of impact sensitivity under the conditions that are imposed by the test procedure.

Preparation of LOX/LN_2 Mixtures

The LOX/LN_2 mixtures used for the tests reported herein were prepared by weighing the required amount of LOX and adding the necessary quantity of LN_2 to give the desired total weight of mixture. The liquid nitrogen was added to the LOX, and the mixture was stirred with a precooled spatula.

To check the accuracy of the composition of the mixtures, analyses were made of control mixtures using an Orsat gas analyzer and a phase diagram to determine the composition of the liquid. In each instance, the mixture was analyzed after being made up for varying periods of time to determine the effect of boil-off on concentration. The details of the test setup are shown in FIG 1; typical results are given in FIG 2.

Inspection of these data indicates, as expected, that the increase in LOX concentration on standing was greatest for those mixtures containing the smallest percentage of LOX. During the 20-minute period normally required for the testing of 20 samples at any given energy level, the average deviation from the nominal LOX concentration was positive and ranged from less than 2 percentage points for the 50/50 mixture to approximately 3 percentage points for the 20/80 mixture. These deviations would not be expected to influence appreciably the results which were obtained during this investigation.

In addition, analyses were made of the actual mixture used for several test samples. This was done by placing samples in the aluminum test cups which then were placed in a steel tray surrounded by an LN_2 moat. The test mixture was poured into the precooled pan and then analyzed. The test setup is shown in FIG 3, and typical results (FIG 4) agree closely with those obtained from the Dewar analyses.

Materials Tested

The materials which were selected for testing represent a wide range of physical and chemical properties; however, each previously had been found to be impact sensitive in 100 percent LOX at 10 kg-m. The materials and the thicknesses in which they were tested were as follows:

Material	Thickness (Inches)
Micarta	0.063
Hexcell 91 LD Honeycomb	0.25
HT-424 Adhesive	0.013
FM-1000 Adhesive	0.010
E-Bond Rubber Sealant H1018	0.050
Hexcell Polyurethane Insulation 1414-2	0.250
Redwing Silicone Rubber	0.063
5Al-2.5Sn Titanium Alloy	0.063
Mylar	0.001
Magnolia 7015-1	0.25
CPR 20 Insulation	0.25
CPR 1021-2 Foam	0.25
HRP Honeycomb filled with CPR 1021-2 Foam, Glued to 2016-T6 Aluminum	0.44

Results

The results are presented graphically in FIG 5 through 17. Each plotted point represents the percentage of reactions in at least 20 tests.

Results for most of the materials indicate that relatively large proportions of LN_2 were required to reduce the reaction frequencies or to increase the threshold energy levels appreciably. This is further demonstrated in FIG 18 in which the observed threshold levels (the energy levels corresponding to a zero reaction frequency) are plotted as a function of the mixture ratio. Inspection of the results indicates that the rate and extent of decrease vary widely and probably are characteristic of the individual materials. However, addition of 8 percent of LN_2 to the LOX generally resulted in a decrease in the threshold energy level of roughly 1 kg-m.

Even highly sensitive materials apparently did not react in 20/80 mixtures (liquid air). However, reactions were noted with several materials at only slightly greater LOX concentrations (30/70), and it is possible that other materials would react with liquid air under suitable stimuli.

Mylar, which gave a reaction frequency of only 20 percent in LOX at 10 kg-m remained slightly sensitive at a LOX concentration of only 30 percent at an energy level of 8 kg-m.

The relatively large quantities of LN_2 required to desensitize most materials indicate that desensitization is due to a dilution or inerting effect rather than to any tendency of the LN_2 to chemically or otherwise inhibit the reaction.

HRP Honeycomb filled with CPR-1021-1 foam was impact sensitive down to 3 kg-m when tested in a 30/70 mixture. It is interesting to note that the CPR-1021-2 foam tested alone was not sensitive at 10 kg-m in a 80/20 mixture. The results of these tests indicate the difficulty in predicting the sensitivity of a composite material from the sensitivity of its components.

CONCLUSIONS

The results of this investigation indicate the following:

1. The sensitivity of most materials to impact with LOX is decreased by dilution of the LOX with LN_2.

2. The extent of dilution necessary to effect an appreciable decrease in reactivity is large; thus, although all materials tested were insensitive in liquid air (20 percent LOX), several were sensitive at 30 percent LOX, and the sensitivity of some materials at 50 percent LOX approached that in pure LOX.

3. The mechanism of the process probably involves a simple inerting action.

4. The sensitivity of a composite material is not a simple function of the sensitivities of its individual components.

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1. Key, C. F. and Gayle, J. B.: Preliminary Investigation of Fire and Explosion Hazards Associated with S-II Insulation. NASA TM X-53144, October 2, 1964.

2. Lucas, William R. and Riehl, Wilbur A.: An Instrument for Determination of Impact Sensitivity of Materials in Contact with Liquid Oxygen. ASTM Bulletin, February 1960, pp. 29-34.

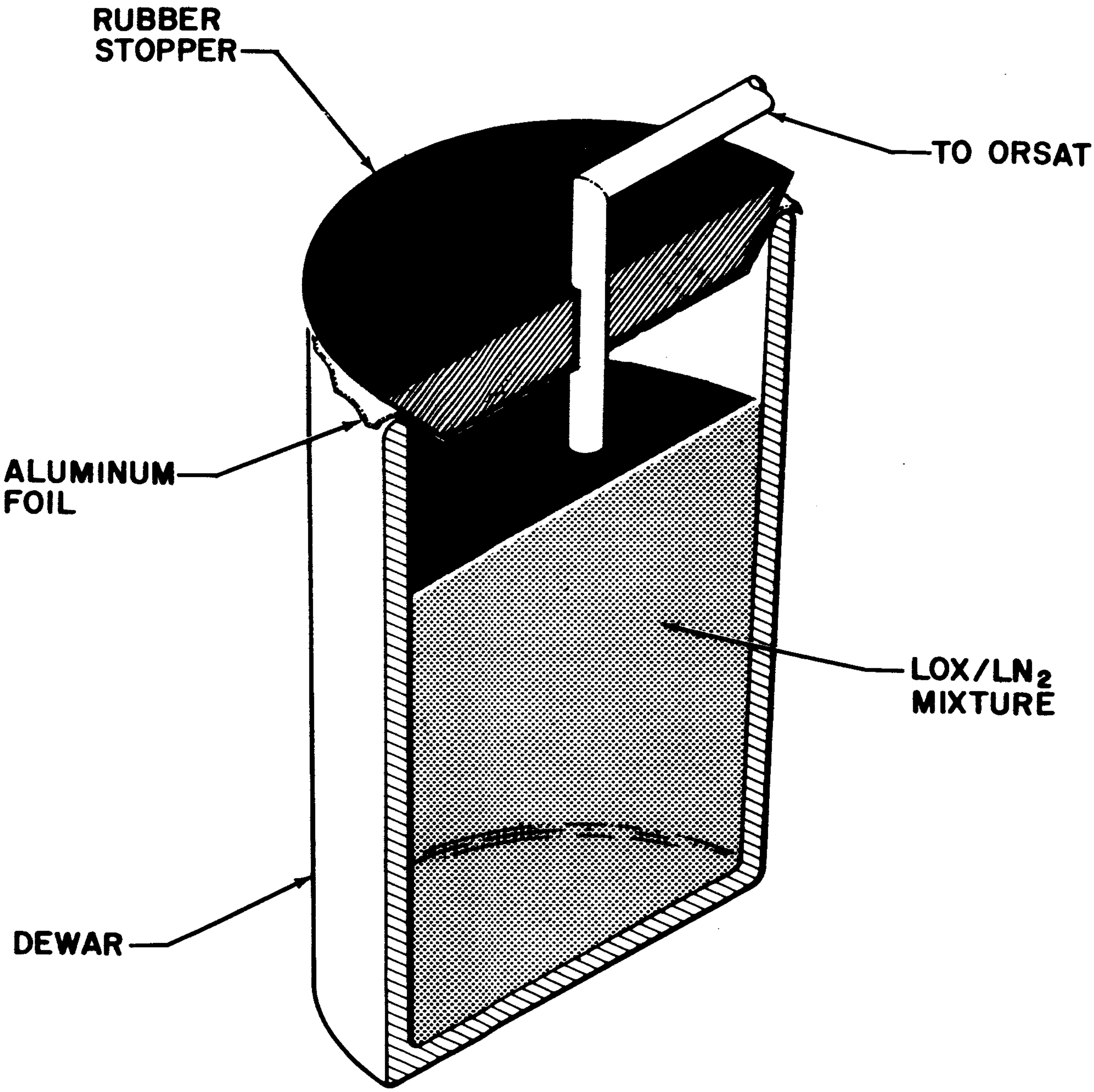


FIGURE 1. TEST SETUP FOR DETERMINING CHANGE IN COMPOSITION OF LOX-LN$_2$ MIXTURES IN DEWAR

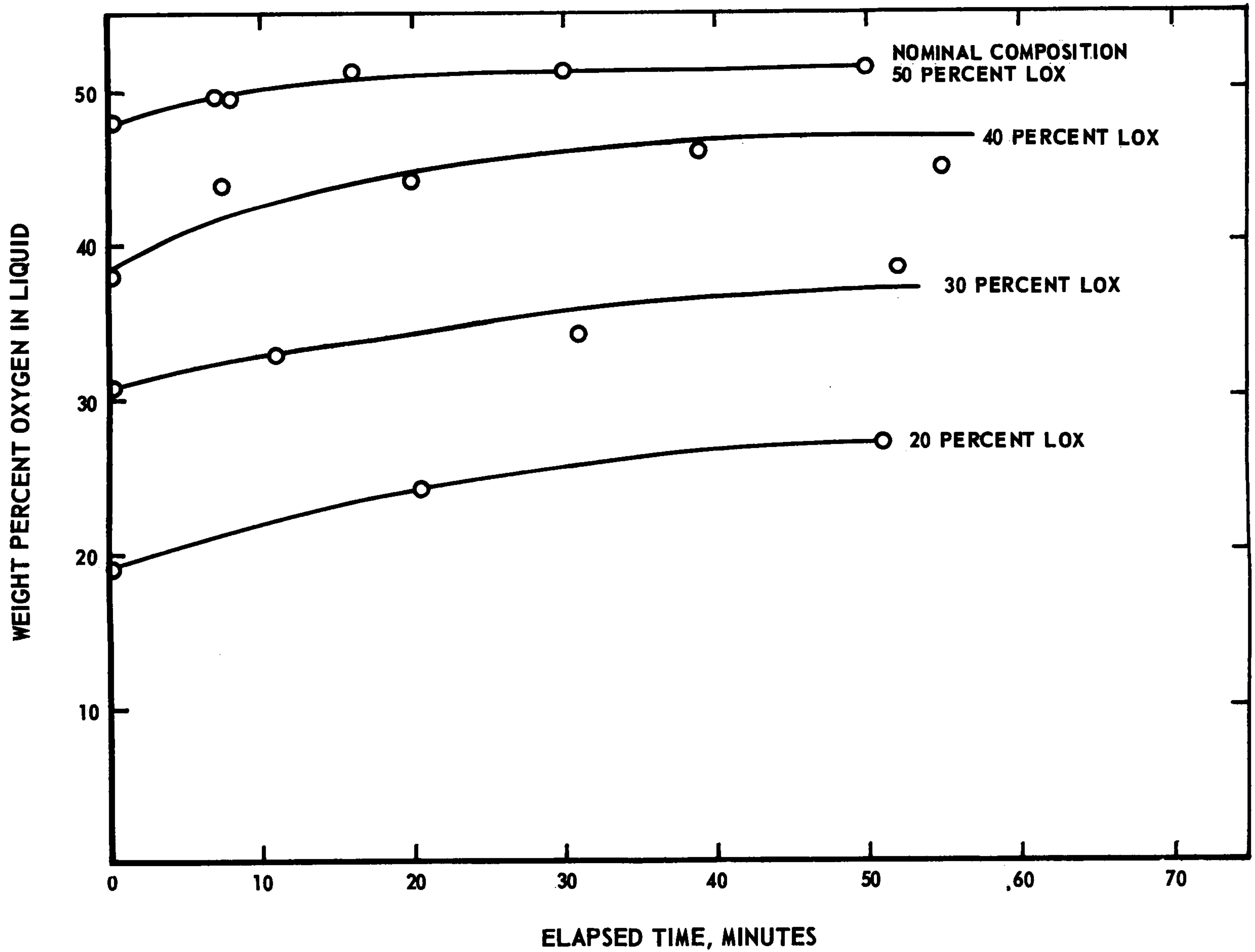


FIGURE 2. ORSAT ANALYSES OF LOX/LN_2 MIXTURES IN DEWAR

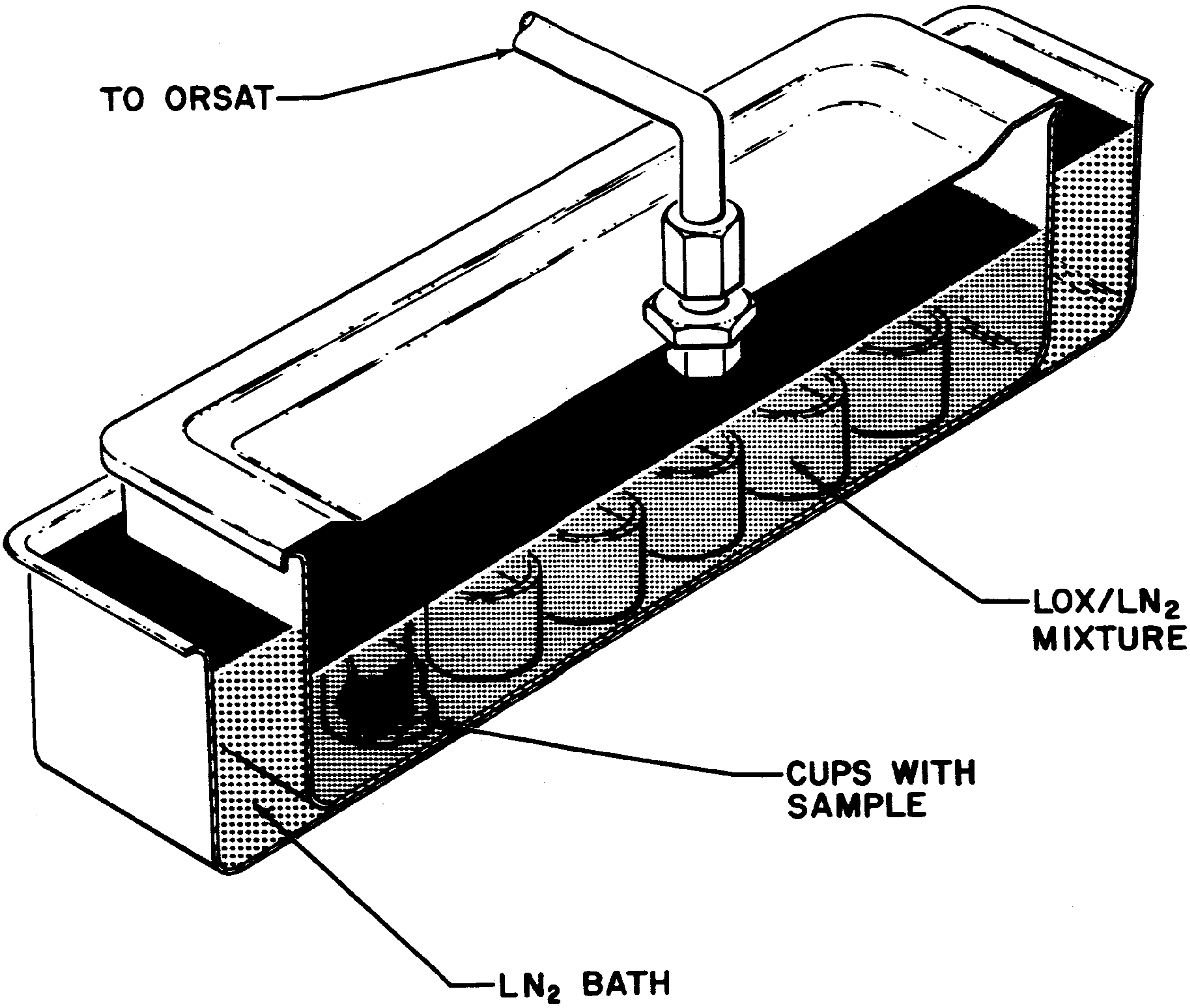


FIGURE 3. TEST SETUP FOR DETERMINING CHANGE IN LOX/LN_2 MIXTURES WITH SAMPLES

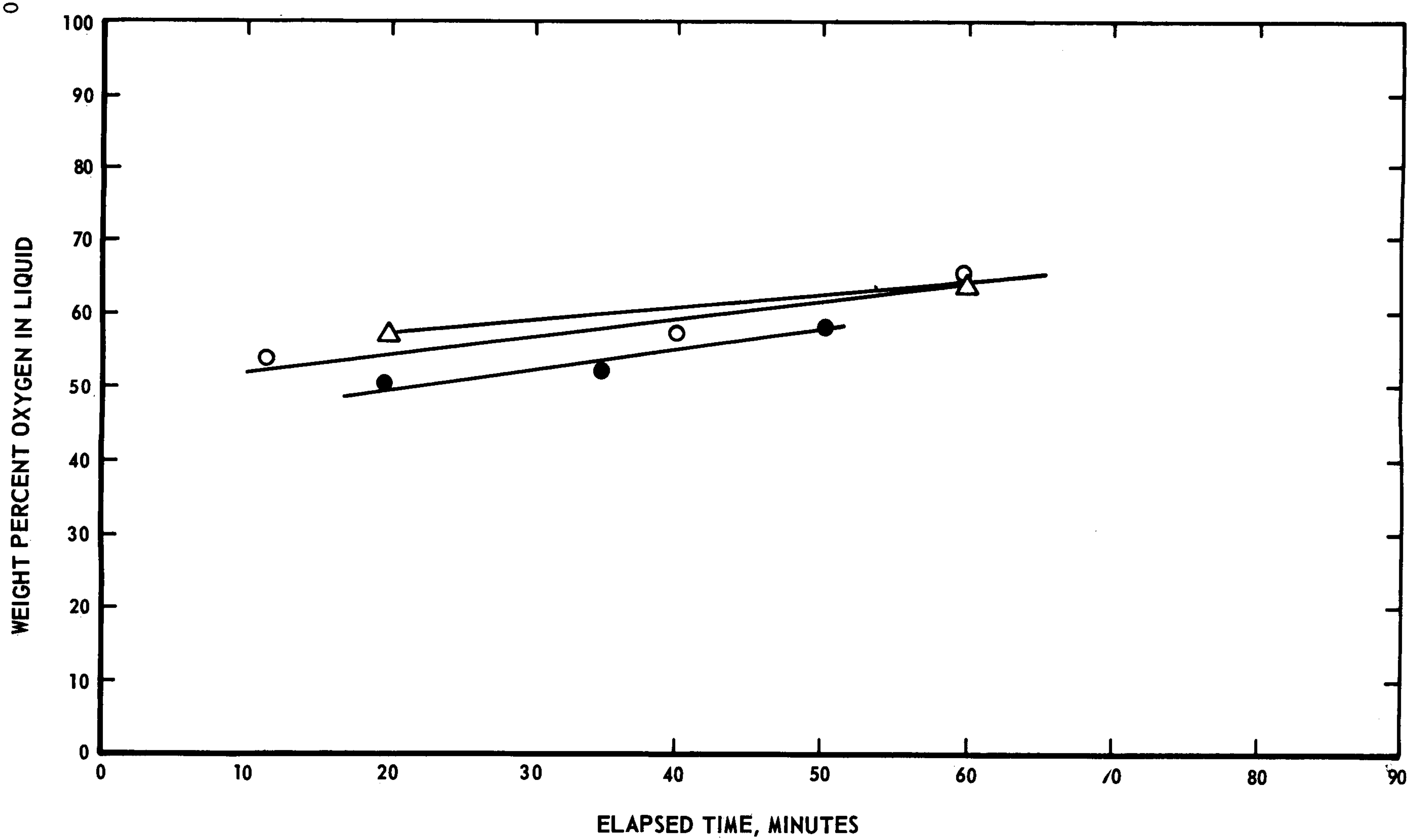


FIGURE 4. ORSAT ANALYSIS OF LOX/LN_2 MIXTURES WITH SAMPLES (NOMINAL COMPOSITION, 50% LOX, 50% LN_2)

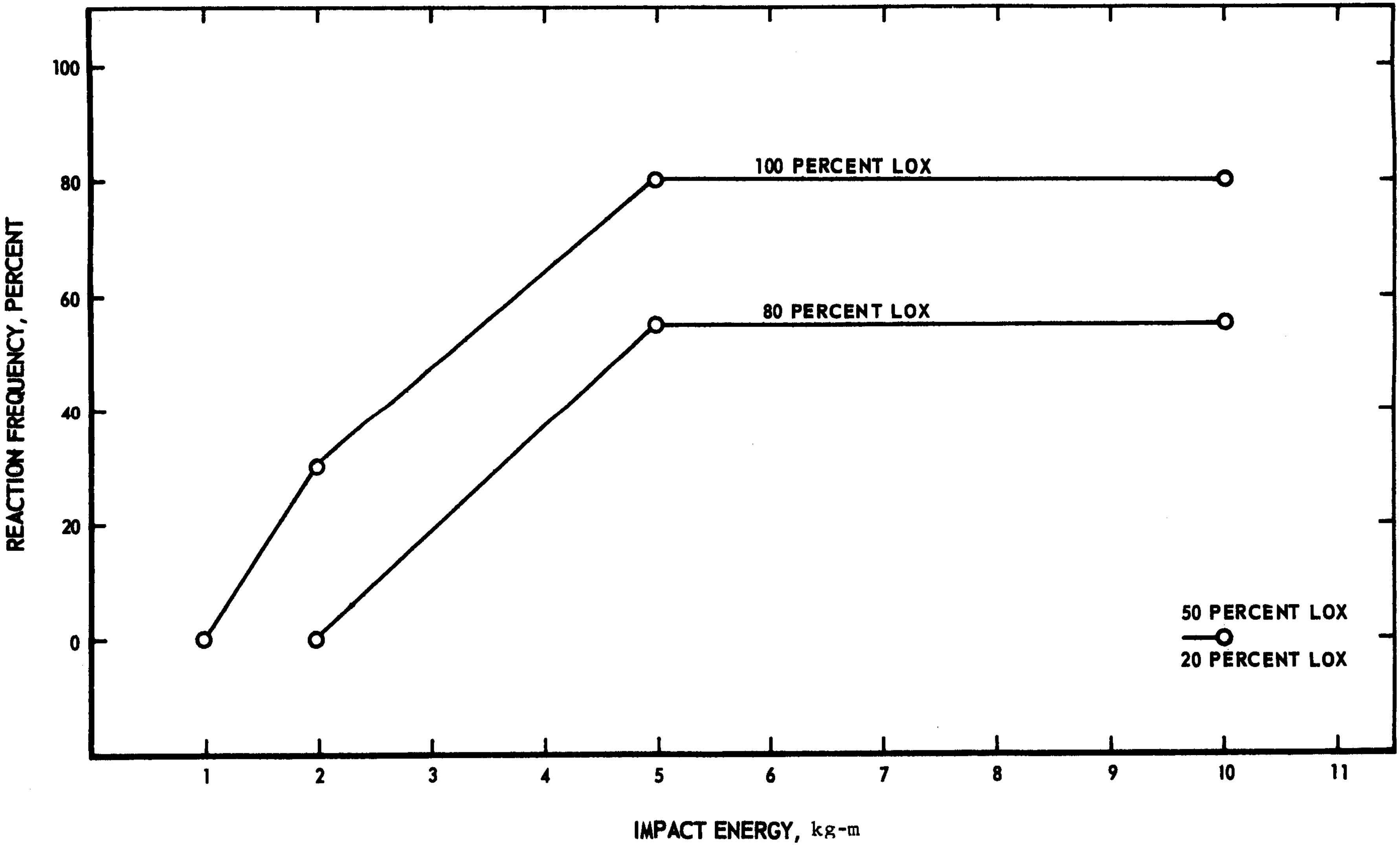


FIGURE 5. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF MICARTA

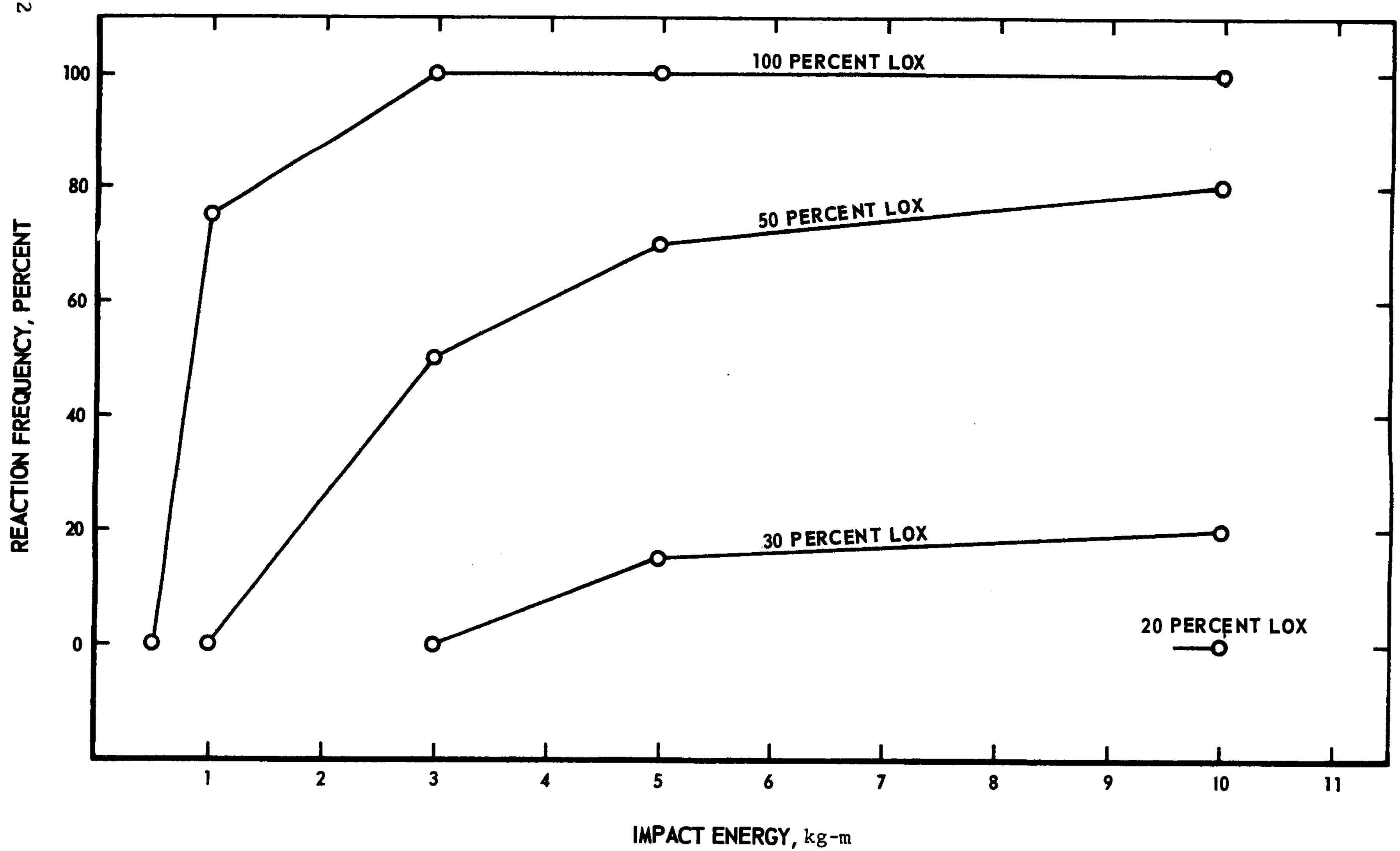


FIGURE 6. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF HEXCELL 91LD HONEYCOMB

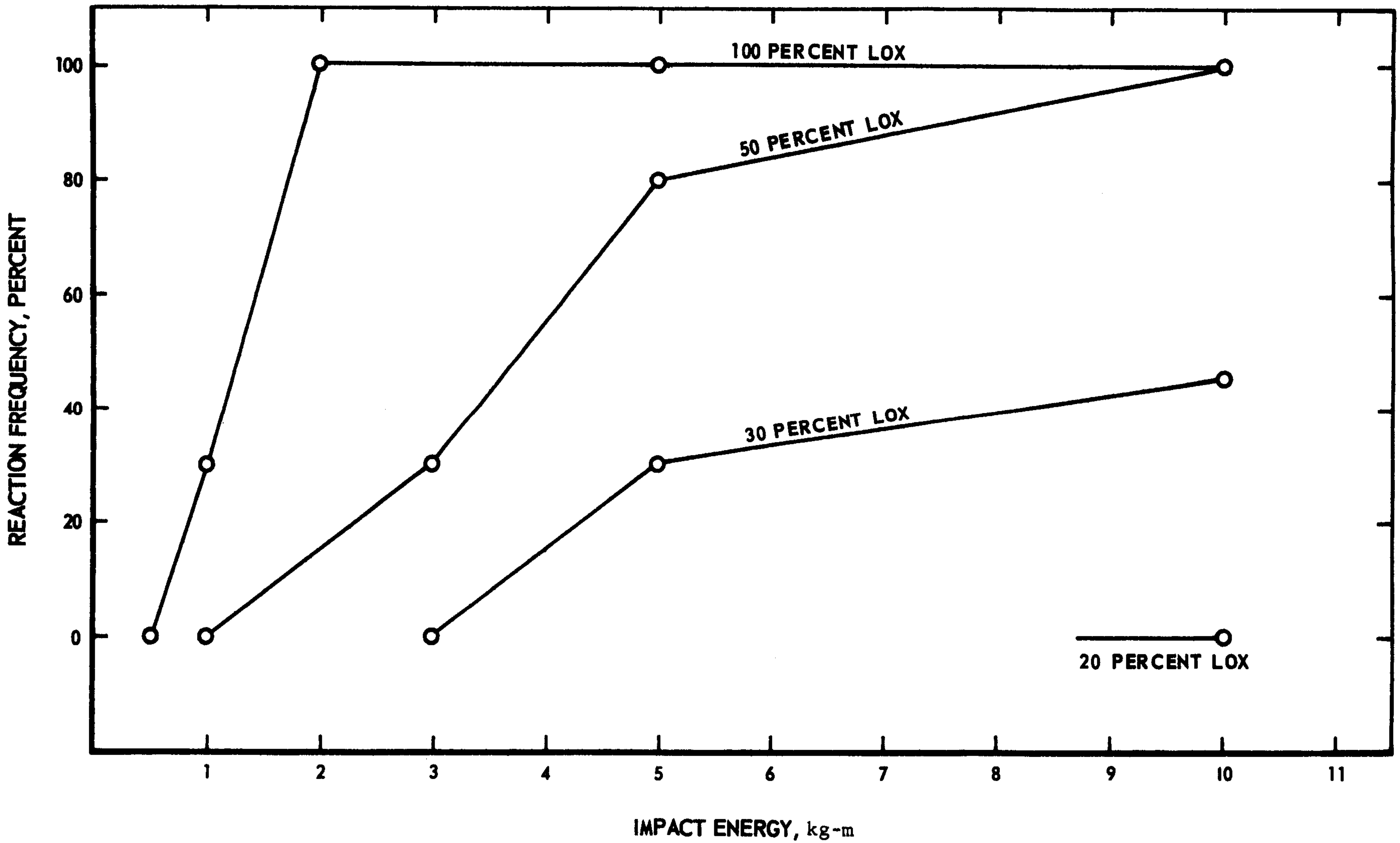


FIGURE 7. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF HT-424 ADHESIVE

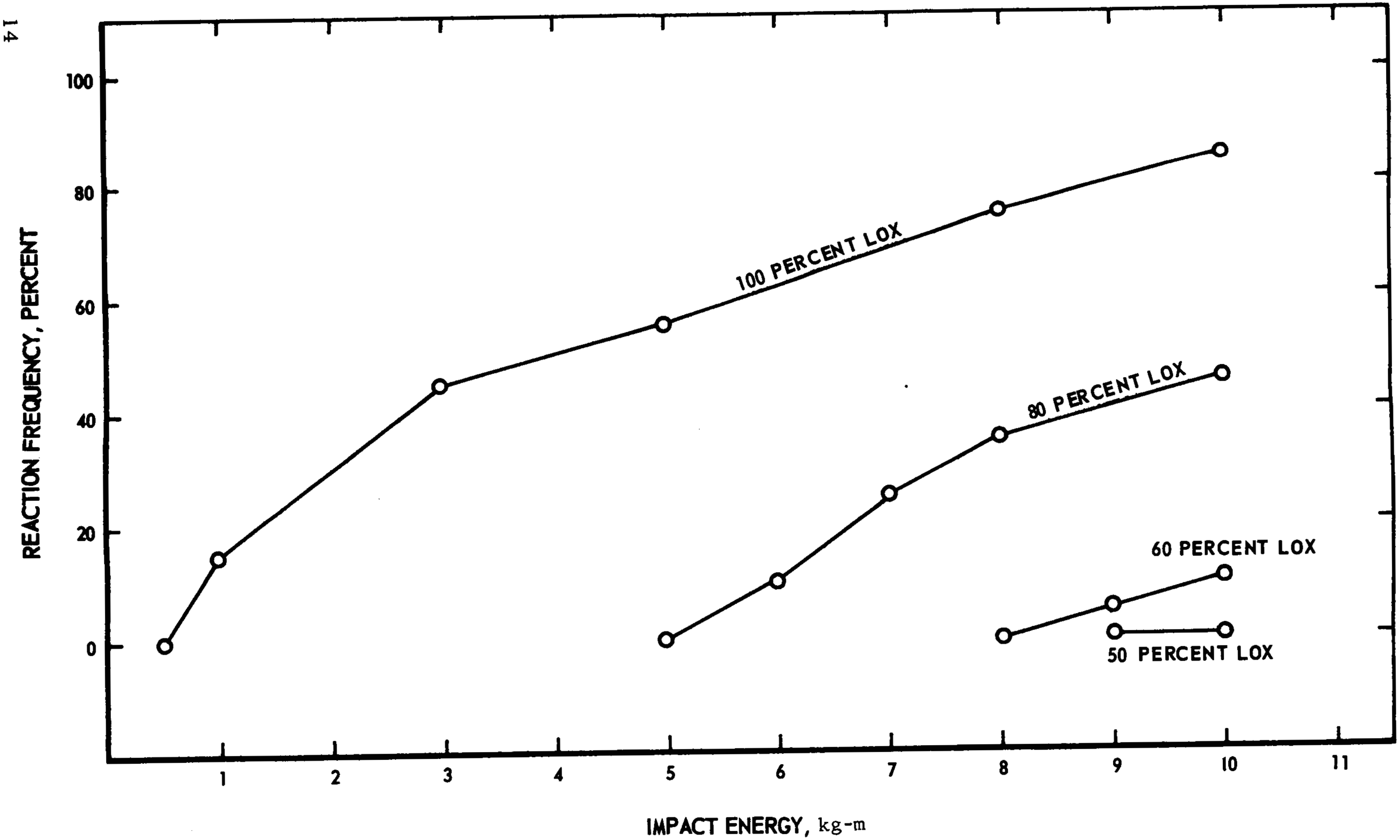


FIGURE 8. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF NYLON EPOXY ADHESIVE FM-1000

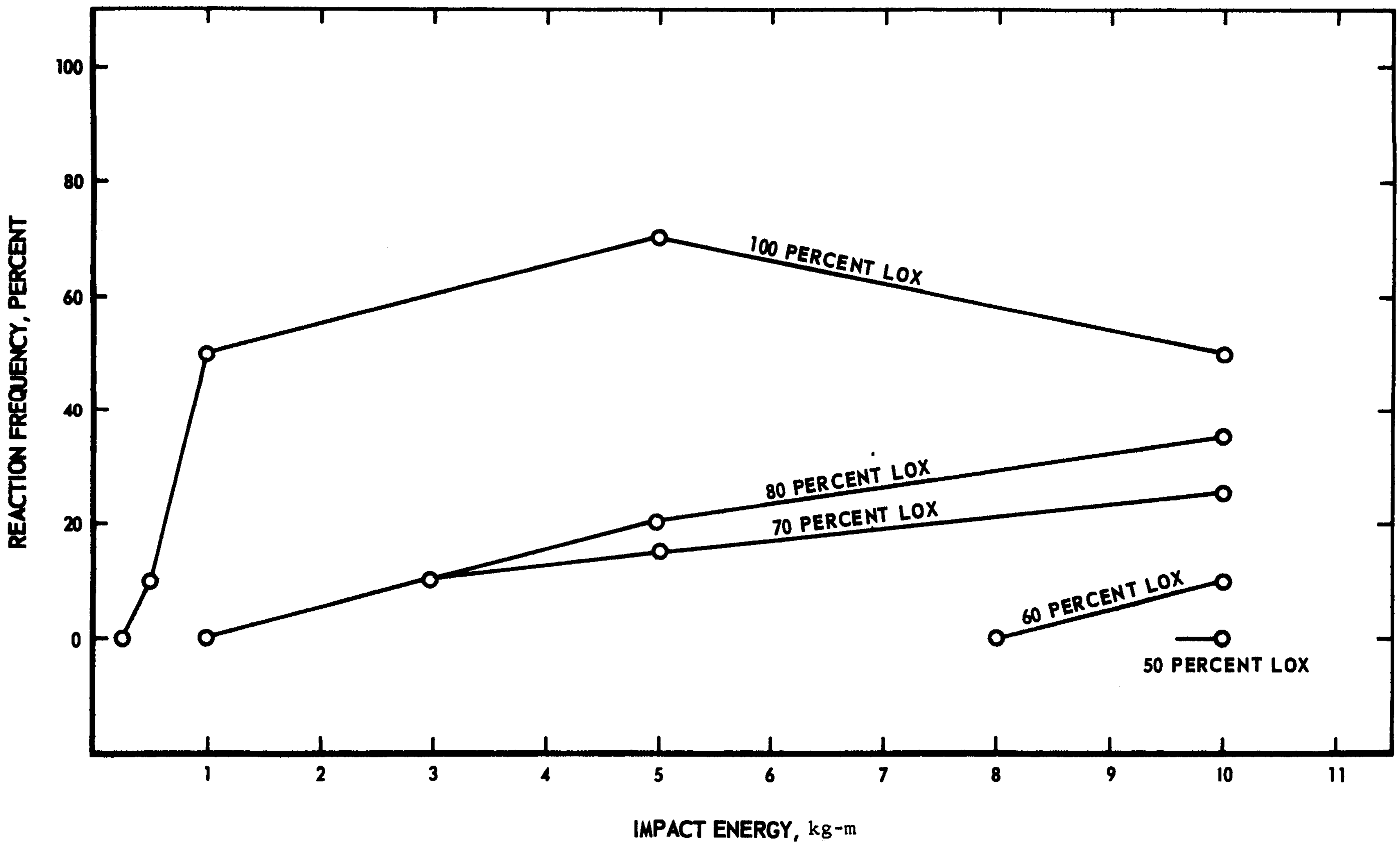
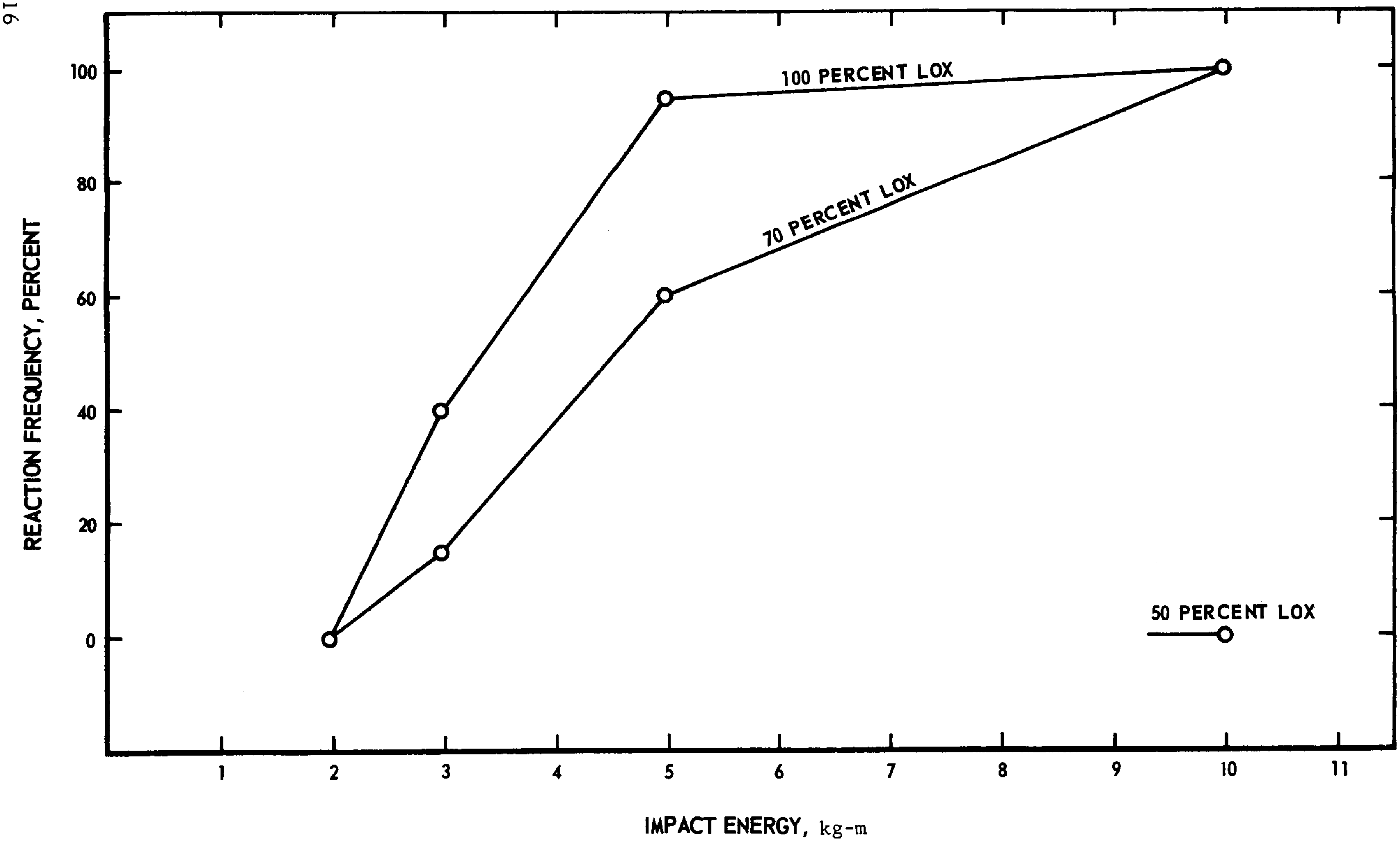


FIGURE 9. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF E-BOND RUBBER SEALANT 1018



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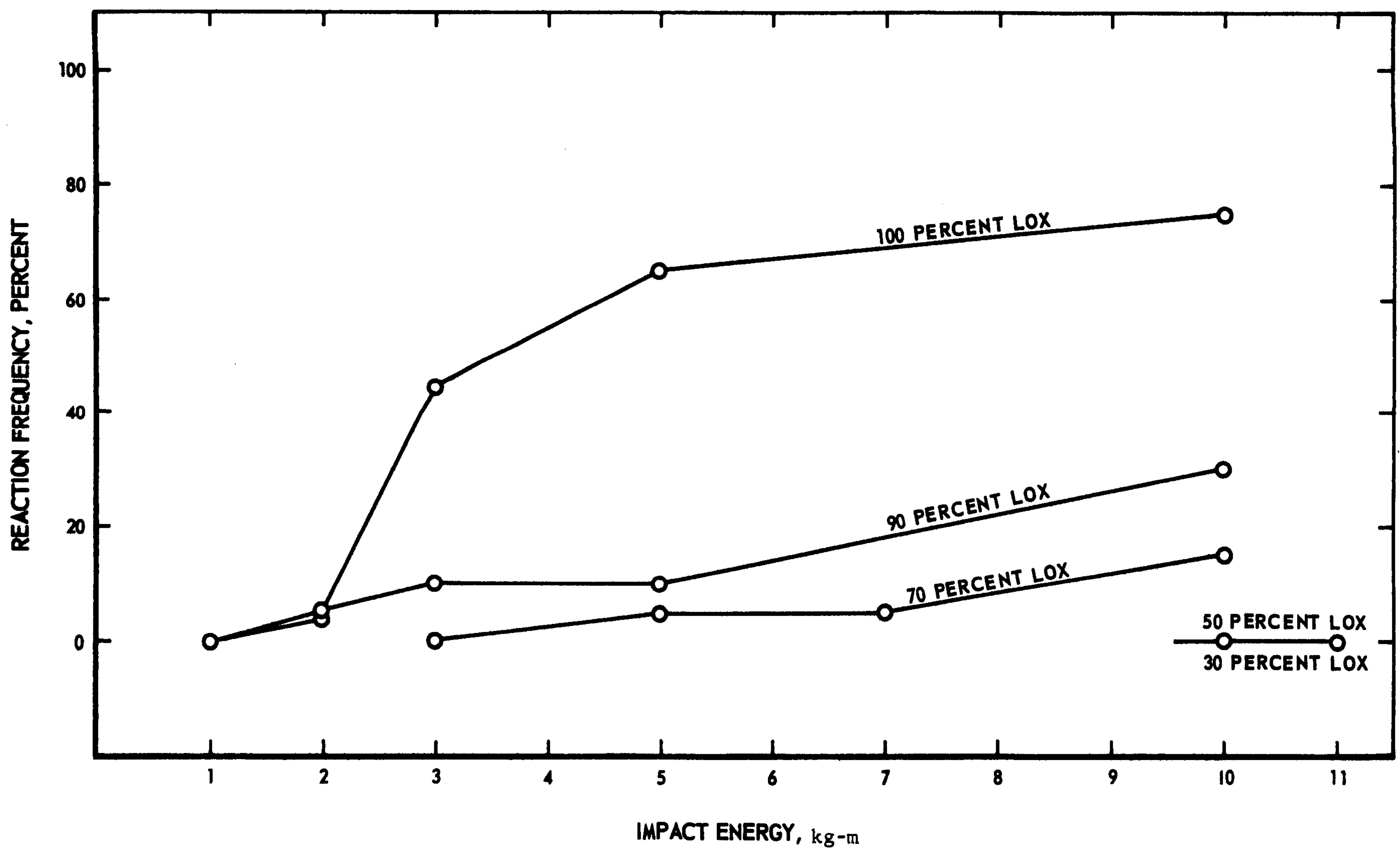

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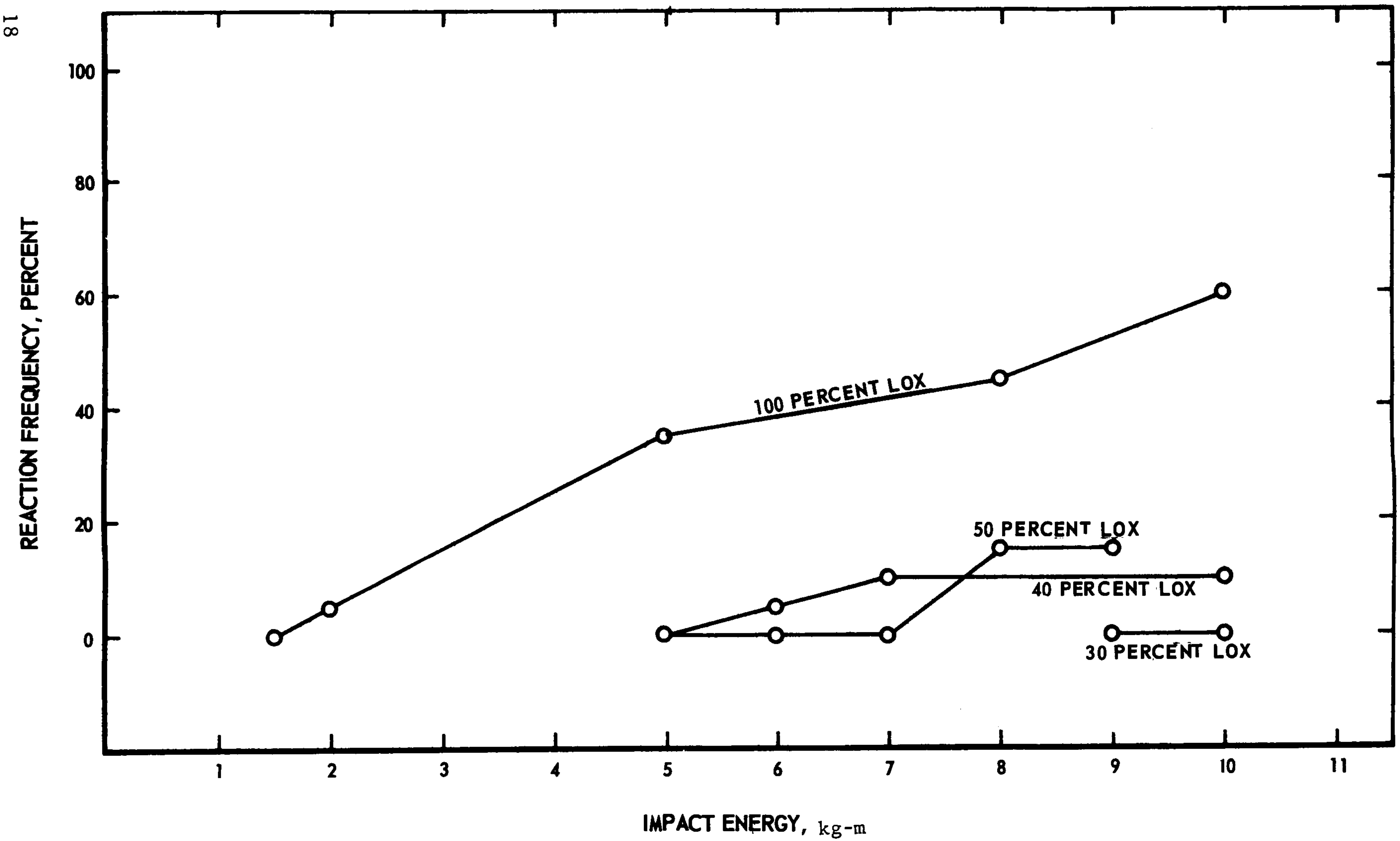

FIGURE 12. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF 5Al-2.5Sn TITANIUM ALLOY, 0.063-INCH THICK

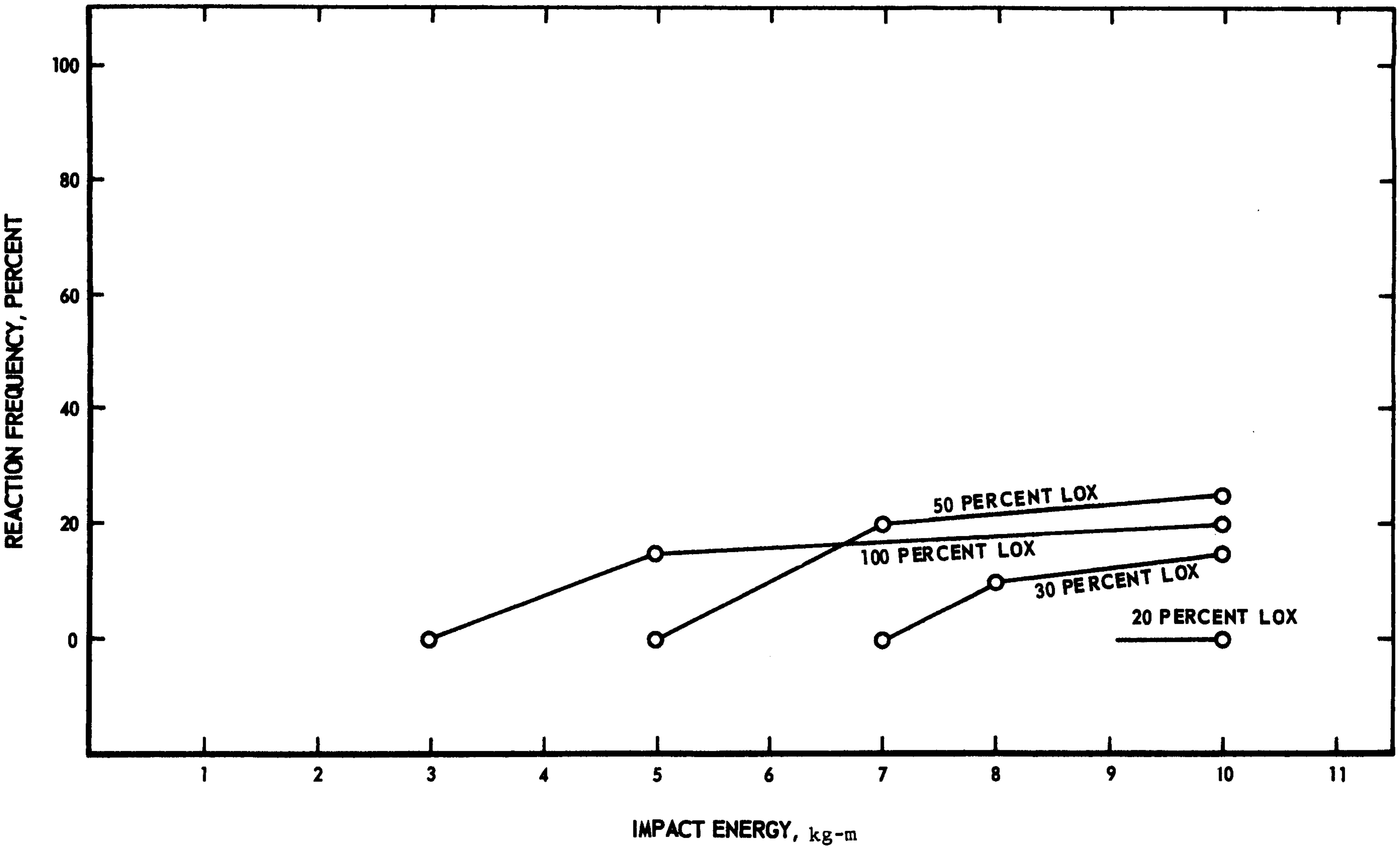

FIGURE 13. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF MYLAR, 1-MIL THICK

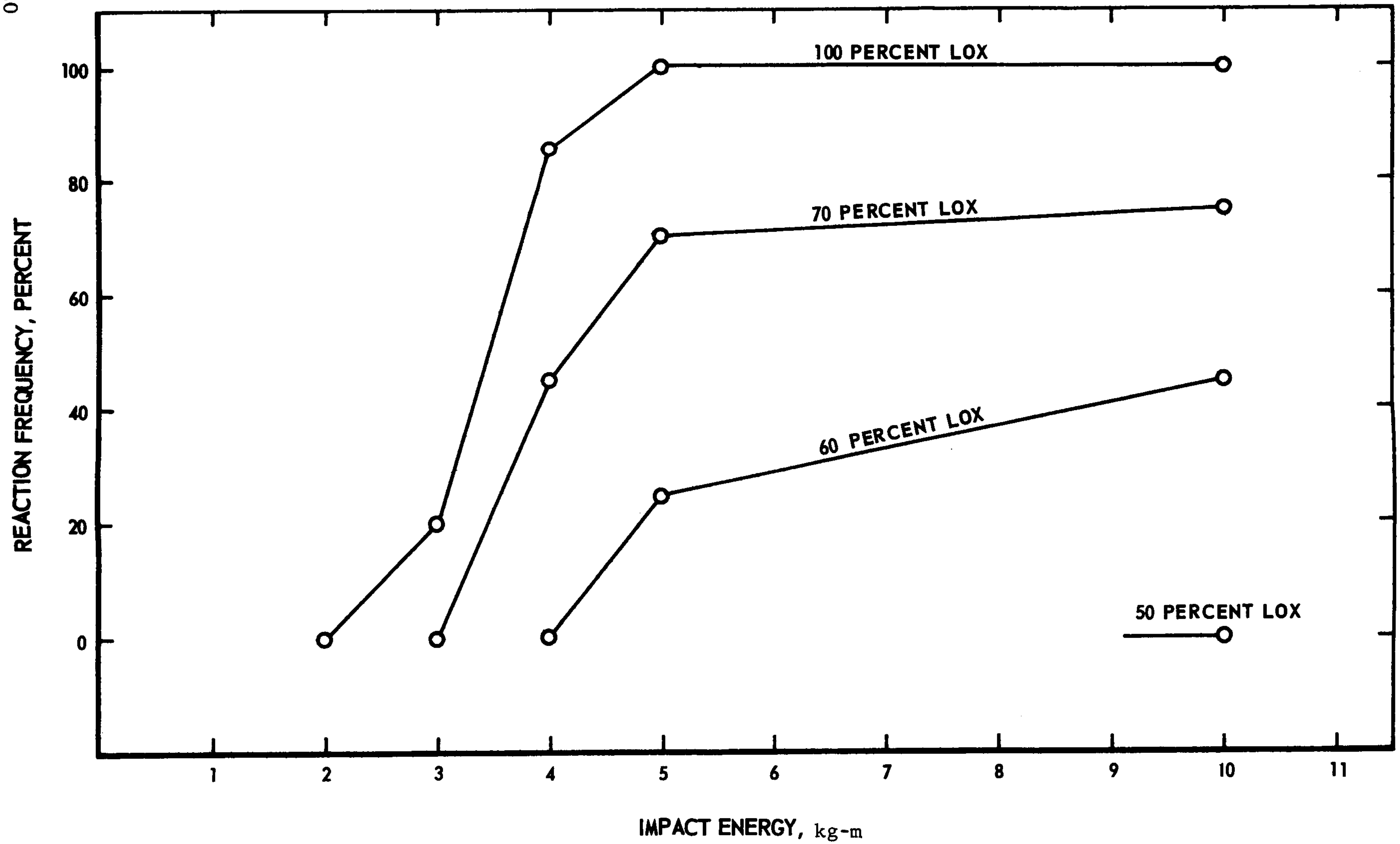

FIGURE 14. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF MAGNOLIA FOAM 7015-1

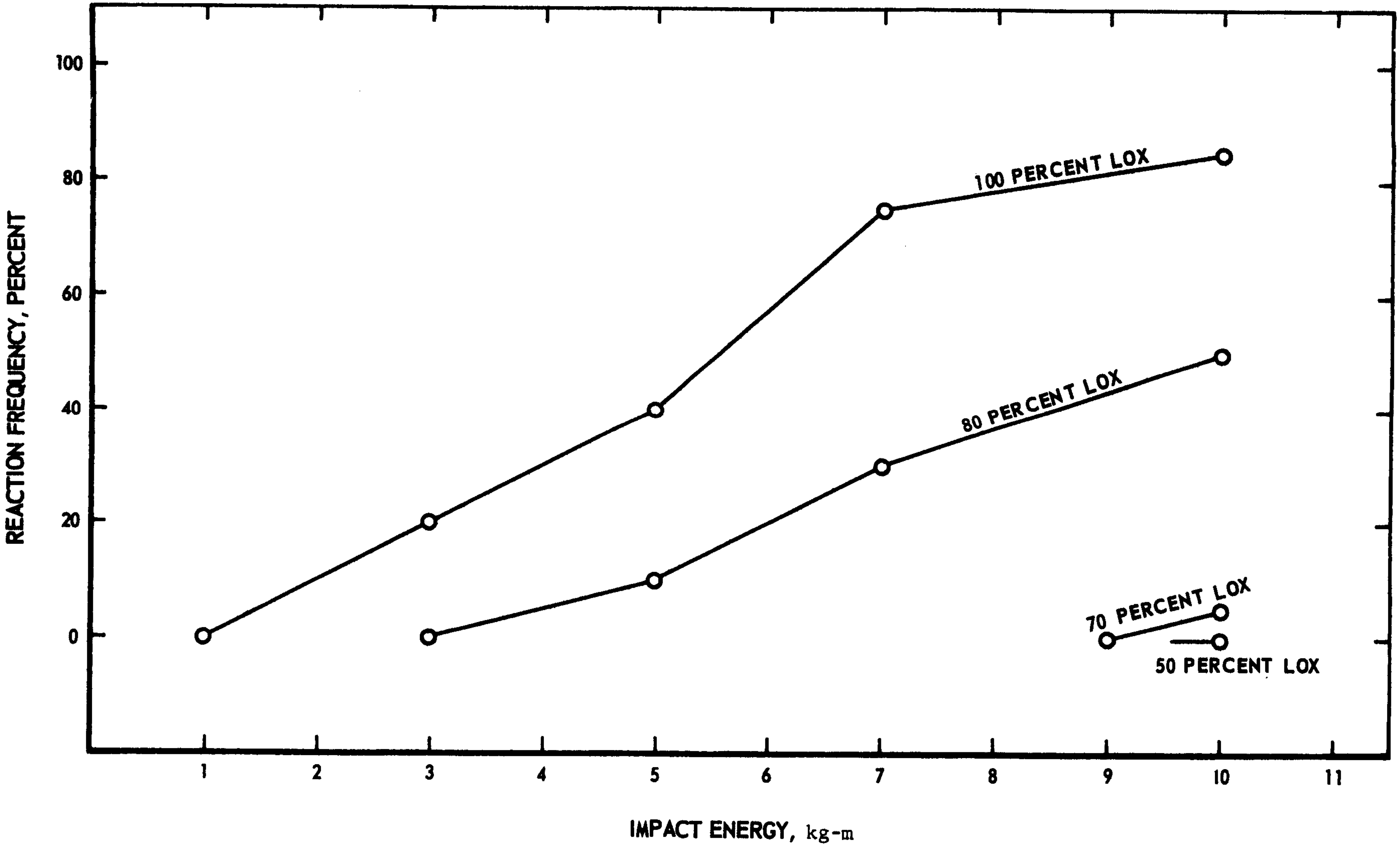

FIGURE 15. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF CPR-20 INSULATION DENSITY - 4#/FT^3

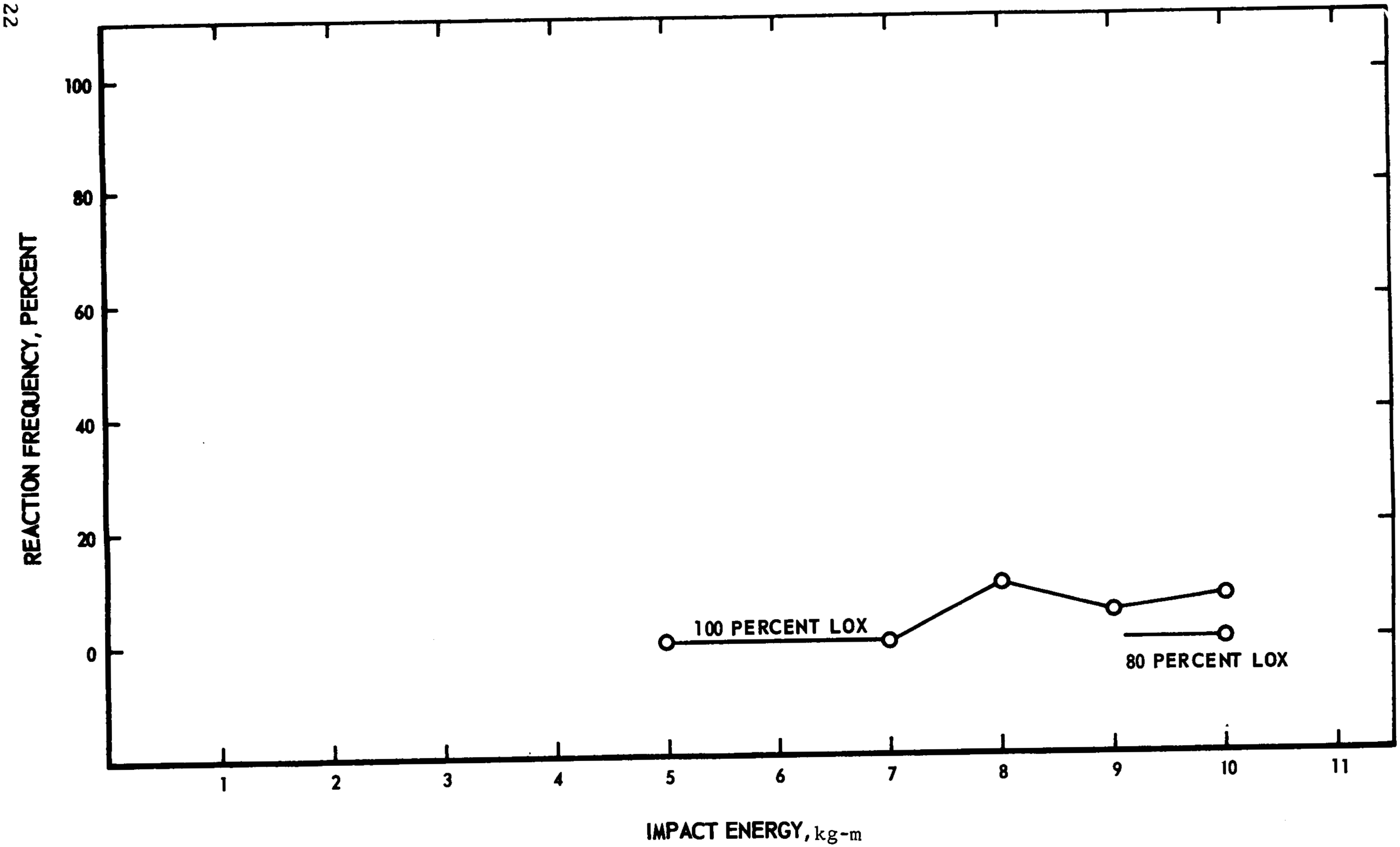

FIGURE 16. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF CPR 1021-2 FOAM

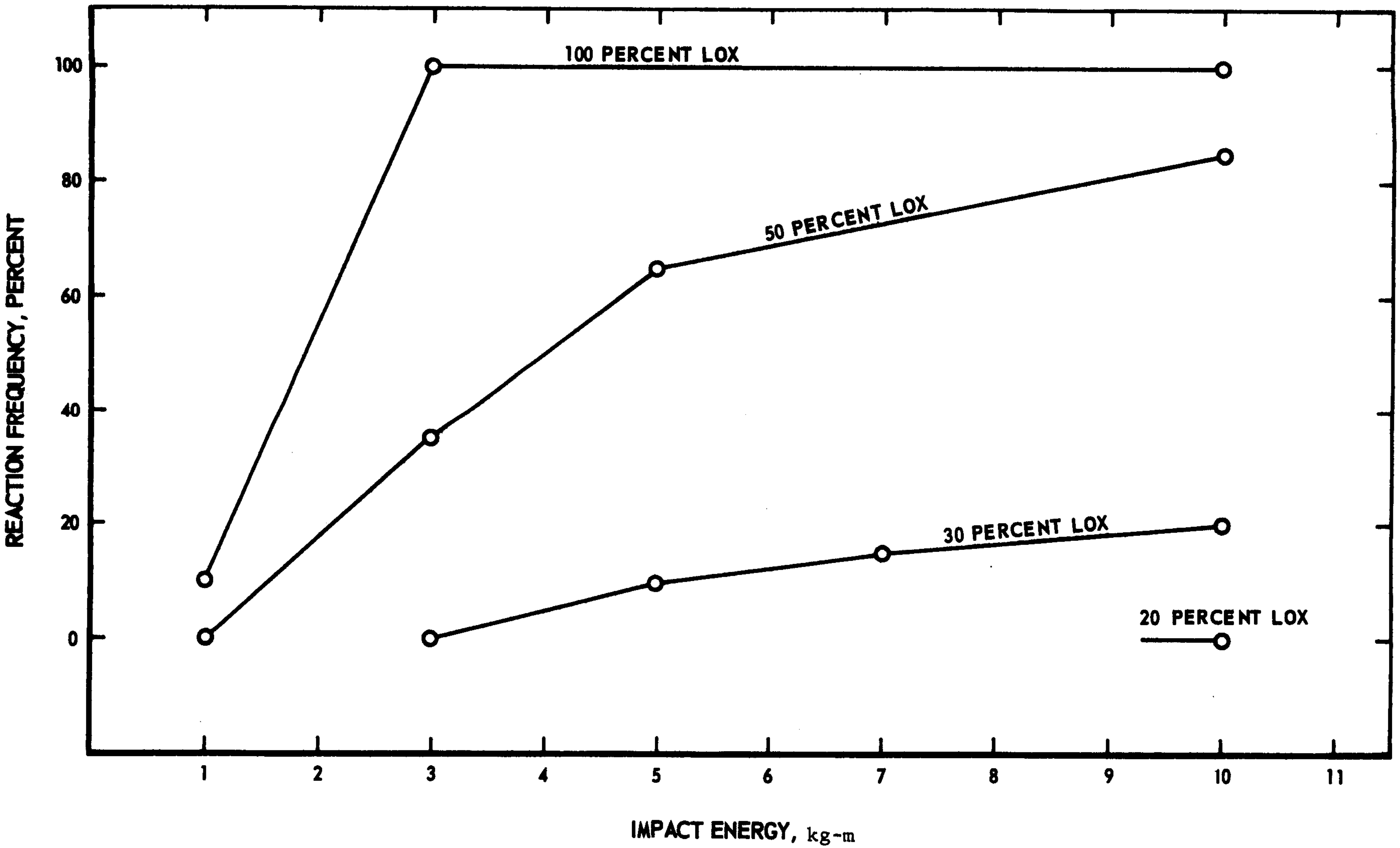

FIGURE 17. EFFECT OF LN_2 DILUTION ON LOX IMPACT SENSITIVITY OF HRP HONEYCOMB FILLED WITH CPR 1021-1 FOAM BONDED TO 2016-T6 ALUMINUM

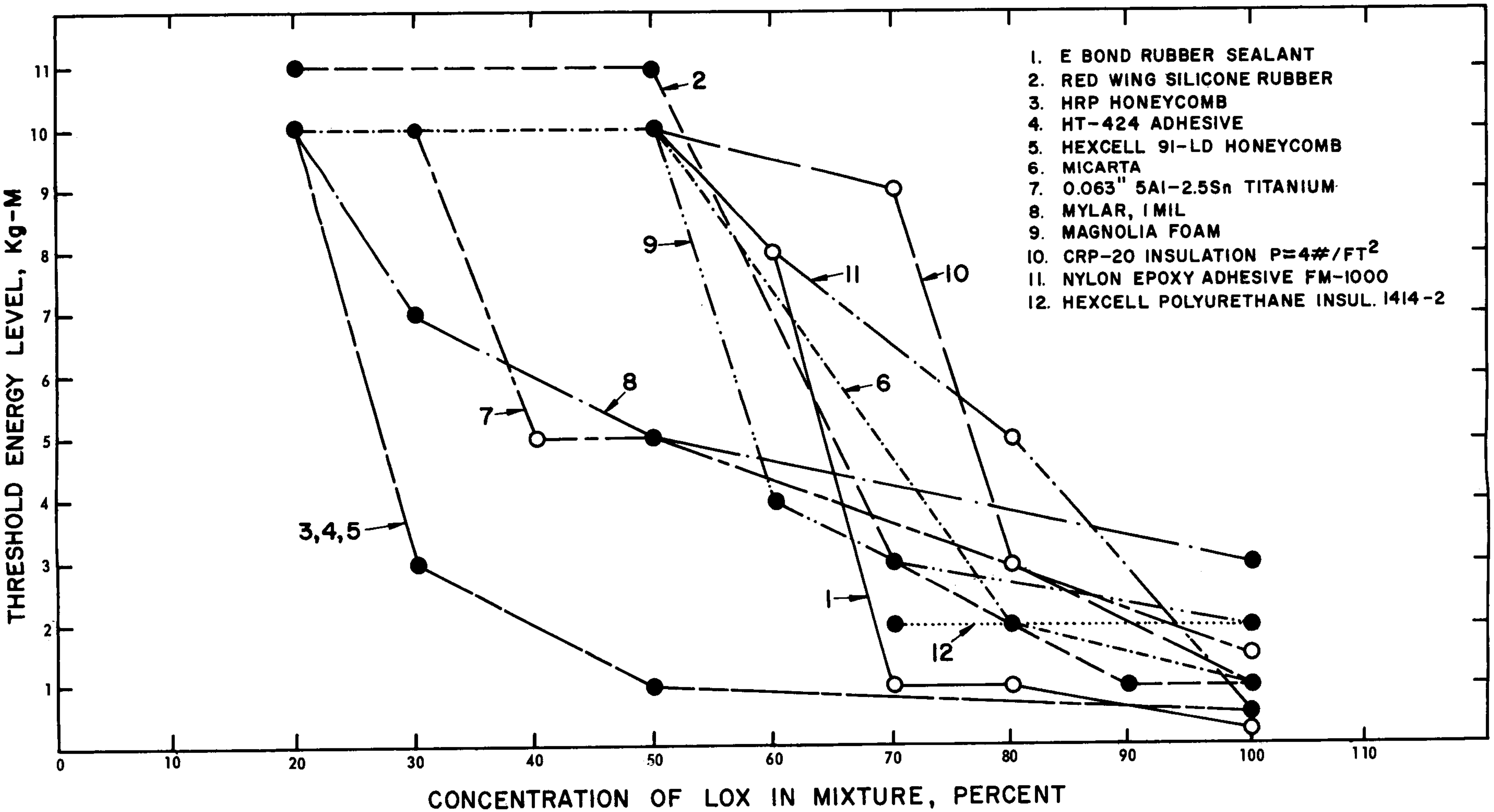

FIGURE 18. EFFECT OF LN_2 DILUTION ON THRESHOLD VALUES FOR VARIOUS MATERIALS

February 15, 1965 APPROVAL NASA TM X-53208

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This document has also been reviewed and approved for technical accuracy.

W. Riehl

W. RIEHL
Chief, Chemistry Branch

W. R. Lucas

W. R. LUCAS
Chief, Materials Division

Emil A. Hellebrand

F. B. CLINE
Acting Director, Propulsion and Vehicle Engineering Laboratory

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